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Mössbauer Hydrides

$Mg_2Ni_{0.75}Fe_{0.25}$

$MMNi_{4.15}Fe_{0.85}$

Mischmetal

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Mössbauer measurements have been made on $Mg_2Ni_{0.75}Fe_{0.25}$ and $MMNi_{4.15}Fe_{0.85}$ and their hydrides. Experimental results demonstrated electronic properties similar to those of other R-E-transition intermetallics. Magnetization, SEM and X-ray diffraction measurements were also obtained. Magnetization and Mössbauer experiments rendered unique results for the mishmetal and showed evidence of a magnetic transition.

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Mössbauer Investigation of $Mg_2Ni_{.75}Fe_{.25}$ and Its Hydride

FINAL REPORT

Frederick Oliver

Windsor Morgan

11/15/88

U.S. ARMY RESEARCH OFFICE

DAAL03-8G-G-0001

MORGAN STATE UNIVERSITY

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SUMMARY OF RESULTS

The research completed under the auspices of Grant DAA LO3-86-6-0001 was very successful. Results were published and presentations were made at national scientific conferences.

A. The work we now review highlights some of our more important efforts:

I. Studies on $MMNi_{4.15}Fe_{0.85}$

We investigated the magnetic and structural properties of a mischmetal-nickel-iron intermetallic and its hydride, using magnetization, Mössbauer, X-ray diffraction, and SEM measurements.

Magnetic measurements as a function of temperature were run on the mischmetal and its hydride. Both samples have a Tc near 200 K. It is a ferromagnetic or ferrimagnetic type ordering. At room temperature, above Tc, hydrogenation increased the susceptibility by nearly a factor of two, whereas below Tc, the susceptibility was lowered considerably. Thus, it is very clear that hydrogenation has affected the magnetic moments although the basic magnetic ordering of the material appears to remain unchanged. Tc occurs near 200 K. There was a peak near 95 K and a dip near 60 K in both samples. Also, both samples show hysterisis of M vs H. Moments did not saturate after 1000 Oe.

Mössbauer measurements were made as a function of temperature. The ^{57}Fe spectra yielded a doublet at room temperature and demonstrated a change in magnetic behavior similar to the magnetization results at low temperatures. $MMNi_5$ forms an unstable hydride, requiring impractical high pressures for hydriding and exhibiting a large hysteresis. Partial substitution of Ni atoms with Fe atoms lowers the absorption pressure and reduces hysteresis. X-ray diffraction measurements on our sample showed the intermetallic to be single phase with the hexagonal structure of the $CaCu_5$ type.

Scanning electron microscopy showed a decrease in particle size with hydriding. Cracks due to straining in the material as a result of hydriding were also observed. The magnetic susceptibility of the unhydrided sample was 1078×10^{-6} emu/g at room temperature and increased as the temperature decreased. One sample was alternatively hydrided and desorbed ten times in an attempt to investigate the effects of hydriding. This

sample showed a large increase in the susceptibility with a corresponding change in magnetization at low temperature. Similar effects have been observed in other intermetallic compounds upon hydriding. We attribute the increase in magnetization to the formation of nickel or iron clusters in the sample. Mössbauer measurements on $MMNi_{4.15}Fe_{0.85}$ indicated the sample as being single phase and paramagnetic with two peaks at room temperature having a quadrupole splitting of 1.00 $\pm$ 0.01 $mmsec^{-1}$ and an isomer shift of -.13 $\pm$0.01 mm sec^{-1} with respect to Fe foil. These values are similar to those found for $LaNi_{4.9}Fe_{0.1}$ and $LaNi_4Fe$. Our results coupled with previous measurements seem to indicate that the crystal structure and the environment of the iron have the greatest influence on the electron density and electric field at the Mössbauer active site. It also appears that these parameters are not affected by the element at the rare-earth site since the rare-earths in mischmetal are quite different from those studied previously. Our results also explain why previous measurements of hydrides do not show larger changes at the transition element site as a result of hydriding. This is quite different from Mössbauer results on intermetallics at the rare-earth site which indicate that hydriding is accomplished by the formation of binary rare-earth hydrides.

Measurements on a hydrided sample taken at room temperature showed an increase in the isomer shift, representing a decrease in the electron density at the nuclear site. When the hydrogen was desorbed, the isomer shift values were the same as that found for the unhydrided compound. Results from this work will be submitted to the Journal of Less Common Materials for publication.

II. Studies on $Mg_2Ni_{.75}Fe_{.25}$

We successfully hydrided the $Mg_2Ni_{.75}Fe_{.25}$ intermetallic compound. The compound absorbed 1.32 atoms of hydrogen per molecule. The hydrogen could be desorbed around 300 C.

Mössbauer measurements on the compound rendered a single line for the unhydrided compound with an isomer shift of 1.2 mm/s at room temperature. This shows that the compound is in a paramagnetic state. A line width of .689 indicates the possibility of a multiphase compound. This is in agreement with the pressure composition isotherm measurements. Mössbauer measurements down to liquid nitrogen temperatures showed that the sample remained paramagnetic with no change in its magnetic properties.

Magnetic susceptibility measurements showed that the addition of iron increased the magnetic properties. Hydriding the compound caused a tremendous increase in the magnetic properties. The increase was so large that we were not able to make measurements on our system.

X-ray diffraction experiments showed the compound to have the same structure as Mg_2Ni. We plan to submit results of this work for publication.

Mössbauer Measurements	Isomer Shift	Line Width
Room Temp.	1.20 mm/s	0.689 mm/s
90 K	1.37	0.419

Magnetic Susceptibility	
Mg_2Ni	2.79×10^{-6} cgs/g
$Mg_2Ni_{0.75}Fe_{0.25}$	4.97×10^{-4} cgs/g

III. Studies on High-Temperature Superconductors

A collaborative project was initiated on a Mössbauer study of high temperature superconductors. A literature investigation was completed and work begun on Eu based compounds. Our laboratory was successful in making Mössbauer measurements on Eu samples. We expect our laboratory to contiue to follow this avenue of investigation.

1. Oliver, F.W., "Mössbauer Investigations of Intermetallic Hydrides," 42nd Annual National Institute of Science Conference, New Orleans, Louisiana, March 13-17, 1985.

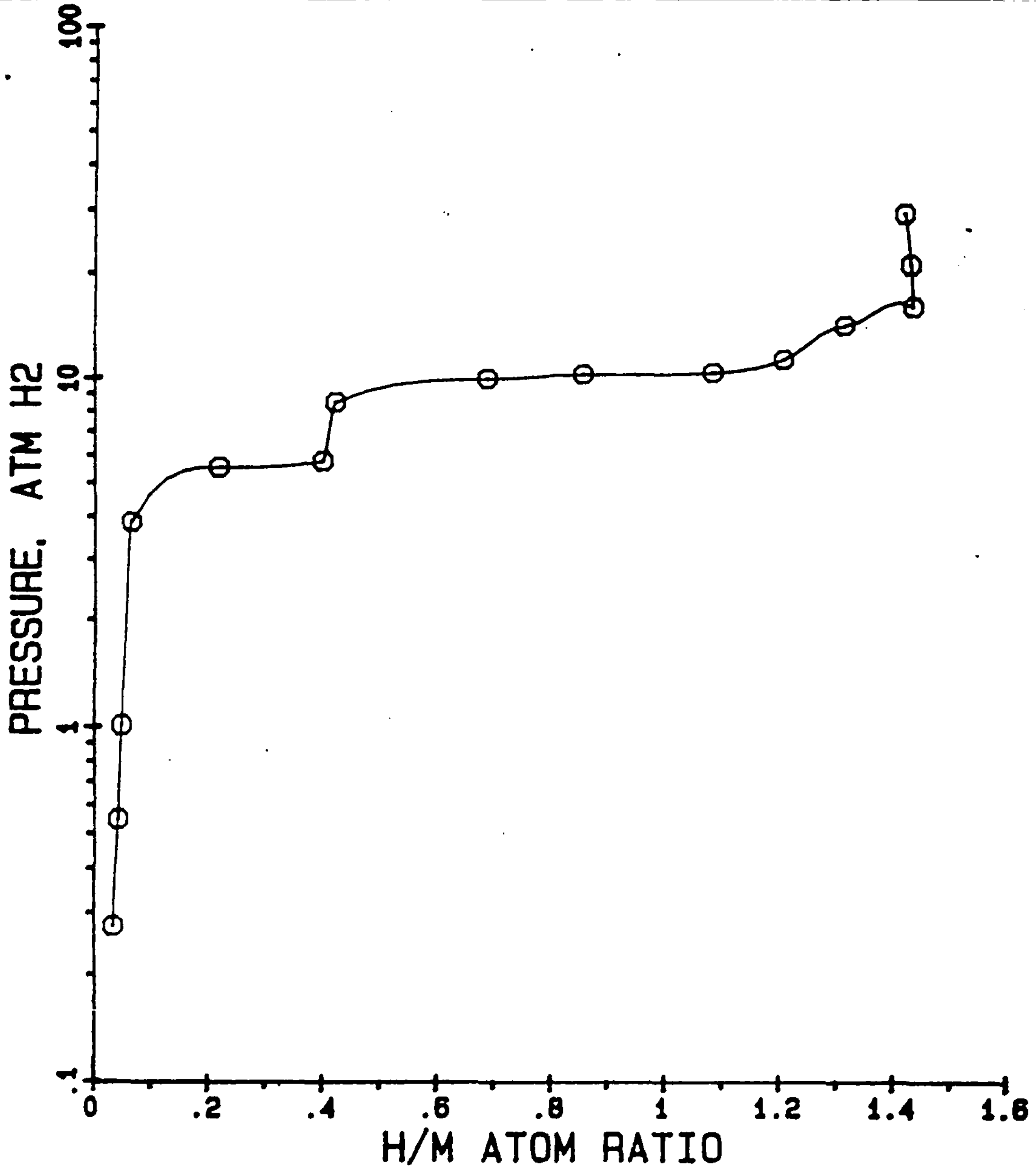
2. Oliver, F.W. et al "Magnetic and Structural Properties of $MMNi_{4.15}Fe_{0.85}$ and Its Hydride," 32nd Annual Conference on Magnetism and Magnetic Materials, Chicago, Illinois, November 9-12, 1987.

3. Oliver, F.W. et al "Magnetic and Structural Properties of $MMNi_{4.15}Fe_{0.85}$ and Its Hydride." Jour. Appl. Phys. 63, (8) 15 April (1988). P. 3620.

4. Oliver, F.W., "Mössbauer Studies on High Temperature Superconductors," 60th Annual National Technical Association Conference, Chicago, Illinois, July 13-16, 1988.

HYDRIDING EXPERIMENT
SPECIMEN :MGNIFE
MODE :ABSORPTION
TEMPERATURE :350C

ALIQ NO	PH2 ATM.	H/M	TS C
0	-.0000	.0	351.1
1	.273	.0334	352.9
2	.544	.0412	351.0
3	1.003	.0480	350.7
4	3.829	.0640	350.7
5	5.523	.2183	350.5
6	5.745	.3999	351.4
7	8.452	.4227	351.3
8	9.903	.6882	351.3
9	10.212	.8571	351.4
10	10.337	1.0848	351.6
11	11.303	1.2085	351.9
12	14.244	1.3151	352.4
13	16.085	1.4353	350.6
14	21.073	1.4305	350.1
15	29.312	1.4209	350.2



PRESSURE, ATM H2
100
10
1
.1
0
.2
.4
.6
.8
1
1.2
1.4
1.6
H/M ATOM RATIO